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Signed

Dated

25 January 2005

Patents Form 1/77

Patents Act 1977  
(Rule 16)



1/77  
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P01/7700 0.00-0329459.2 CHEQUE

**Request for grant of a patent**

(See the notes on the back of this form. You can also get an explanatory leaflet from the Patent Office to help you fill in this form)

19 DEC 2003

LONDON

The Patent Office

Cardiff Road  
Newport  
South Wales  
NP10 8QQ

1. Your reference

REP07649GB

19 DEC 2003

2. Patent application number

(The Patent Office will fill this part in)

0329459.2

3. Full name, address and postcode of the or of each applicant (underline all surnames)

ITM Power Ltd.  
Villa Farm, Jack Haws Lane  
Barnack  
Stamford  
Lincs PE9 3DY

Patents ADP number (if you know it)

If the applicant is a corporate body, give the country/state of its incorporation

8461741001

4. Title of the invention

Hydrophilic Materials used as Ionically Active Membranes

5. Name of your agent (if you have one)

Gill Jennings & Every

"Address for service" in the United Kingdom to which all correspondence should be sent (including the postcode)

Broadgate House  
7 Eldon Street  
London  
EC2M 7LH

Patents ADP number (if you know it)

745002

6. Priority: Complete this section if you are declaring priority from one or more earlier patent applications, filed in the last 12 months.

Country

Priority application number  
(if you know it)

Date of filing  
(day / month / year)

7. Divisionals, etc: Complete this section only if this application is a divisional application or resulted from an entitlement dispute (see note f)

Number of earlier UK application

Date of filing  
(day / month / year)

8. Is a Patents Form 7/77 (Statement of inventorship and of right to grant of a patent) required in support of this request?

YES

Answer YES if:

- a) any applicant named in part 3 is not an inventor, or
- b) there is an inventor who is not named as an applicant, or
- c) any named applicant is a corporate body.

Otherwise answer NO (See note d)

Patents Form 1/77

# Patents Form 1/77

9. Accompanying documents: A patent application must include a description of the invention. Not counting duplicates, please enter the number of pages of each item accompanying this form:

Continuation sheets of this form

Description 4

Claim(s) 1

Abstract -

Drawing(s) -

OL

10. If you are also filing any of the following, state how many against each item.

Priority documents

Translations of priority documents

Statement of inventorship and right to grant of a patent (Patents Form 7/77)

Request for a preliminary examination and search (Patents Form 9/77)

Request for a substantive examination (Patents Form 10/77)

NO

Any other documents (please specify)

11. I/We request the grant of a patent on the basis of this application.

For the applicant

Gill Jennings & Every

Signature

Date 19.12.03

12. Name, daytime telephone number and e-mail address, if any, of person to contact in the United Kingdom

PERRY, Robert Edward

020 7377 1377

## Warning

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## Notes

- If you need help to fill in this form or you have any questions, please contact the Patent Office on 08459 500505.
- Write your answers in capital letters using black ink or you may type them.
- If there is not enough space for all the relevant details on any part of this form, please continue on a separate sheet of paper and write "see continuation sheet" in the relevant part(s). Any continuation sheet should be attached to this form.
- If you have answered YES in part 8, a Patents Form 7/77 will need to be filed.
- Once you have filled in the form you must remember to sign and date it.
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## Hydrophilic Materials Used as Ionically active Membranes.

### Background of the Invention

There are in essence three ways in which a hydrophilic material can be rendered ionically conducting,

- (i) by the inclusion of ionically active sites by *co-polymerisation from solution of ionically active monomers*, acidic (e.g. SO<sub>3</sub>) or anionic (e.g. Vinyl benzyl ammonium chloride); in this case the crosslinked material becomes ionically conducting when hydrated in deionised-distilled (dd water) water.
- (ii) by the inclusion of ionically active sites *by grafting* of ionically active monomers, acidic (e.g. SO<sub>3</sub>); in this case the material (e.g. Nafion) becomes ionically conducting when hydrated in deionised-distilled (dd water) water.

- (iii) by hydration of the hydrophilic material in an ionically active (acid or alkaline) liquid. In this case the hydrophilic material is electrically inert when hydrated in dd water, but conductive when hydrated in the appropriate solution.

Approach (i) above has been patented by ITM Power as GB2380055 together with the novel 'one-step' production route for MEA's and entire stacks, while approach (ii) is well known. However, the potential advantages of item (iii) are not generally appreciated and include:

- (a) control of the equilibrium liquid uptake of the structure which affects the electrical hydraulic and gas transmission properties of the resulting membrane,
- (b) the option to employ low cost hydrocarbon monomer systems,
- (c) the option (subject to the ITM UK Patent) to employ the 'one-step' manufacture process in the production of MEA's and stacks.

### Minimum Liquid Volume Devices

Conventional fuel cells and electrolyzers are constructed using an inert separator flooded with a liquid electrolyte, (a lye) which can be either acid or alkaline to choice. In practice the alkaline system is often favoured because it allows for the use of catalysts which are more effective than in acid solution if Platinum based, or of low cost non-noble metal derivatives (nickel).

Such circulating lye devices are effective but require extensive balance of plant to purify and degas the circulating electrolyte for reuse, they are therefore complex and costly to design, build and operate as well as slow in response to changing loads conditions.

- 5        The advent of PEM solid polymer electrolyte systems typified by the acid chemistry of Nafion has revolutionised the design of fuel cells and electrolyzers but no effective alkaline based membrane has yet been successfully marketed.

#### Electrically Active Hydrophilic Materials

- 10       It has now been demonstrated that crosslinked hydrophilic materials can be formed which when hydrated in suitable acid or alkaline solutions, exhibit electrical properties significantly superior to those of the industry standard solid polymer material Nafion.

- 15       The possible use of hydrophilic materials hydrated in suitable acid or alkaline solutions and operated in a 'minimum liquid' mode is therefore of considerable commercial interest at least until the advent of genuine alkaline SPE materials which operate only in the presence of water.

#### Example 1

- 20       A crosslinked hydrophilic polymer was formed by irradiating a mixture containing 35% acrylonitrile 35% n-vinyl-2-pyrrolidone and 5% allylmethacrylate and 25% deionised distilled water to a dose of 2 megarads in a cobalt 60 source. The resulting solid polymer (reference AN-VP-C25) was divided into two samples. The first was hydrated in water and its electrical conductivity measured using an AC bridge method. The second sample was hydrated in a solution of 10% by weight Toluene Sulphonic acid (TSA) in water, after which its electrical conductivity was measured in the ~~same~~ test cell as used for sample one.

- 25       The results show that the conductivity of the second sample (hydrated in TSA) is greater than that of first sample (hydrated in water) in the ratio of  $5.7:0.07 = 81.4$ .

- 30       Using the same apparatus the conductivity was found to be 36% greater than that of a Nafion 117 membrane.

The second sample was then immersed in an excess of dd water for a period of 14 days and after which its electrical conductivity was again measured,

its 'residua' conductivity was only 15% of its initial conductivity, demonstrating that substantial loss of TSA had occurred. Subsequent measurements demonstrated a continuing decline in conductivity with time of washing in dd water.

## 5 Example 2

A crosslinked hydrophilic polymer was formed by irradiating a mixture containing 24% methyl methacrylate 72% n-vinyl-2-pyrrolidone and 4% allylmethacrylate to a dose of 2 megarads in a cobalt 60 source. The resulting solid polymer (reference MMA-VP1) was divided into two samples. The first was  
10 hydrated in water and its electrical conductivity measured using an AC bridge method. The second sample was hydrated in a solution of 10% by weight Toluene Sulphonic acid (TSA) in water, after which its electrical conductivity was measured in the same test cell as used for sample one.

The results show that the conductivity of the second sample (hydrated in  
15 TSA) is greater than that of first sample (hydrated in water) in the ratio of  $47.6:0.05 = 952$ .

Using the same apparatus the conductivity was found to be 13% greater than that of a Nafion 117 membrane.

The second sample was then immersed in an excess of dd water for a  
20 period of 14 days and after which its electrical conductivity was again measured, its 'residua' conductivity was only 12% of its initial conductivity, demonstrating that substantial loss of TSA had occurred. Subsequent measurements demonstrated a continuing decline in conductivity with time of washing in dd water.

25 These examples demonstrate that the presence of an ionic conductor (10% TSA) renders the hydrated crosslinked copolymer electrically conducting, but that exposure to water removes the active component and ultimately returns the material to a resistive state.

Thus although the material may be used to make a membrane, or an MEA  
30 or a stack the use of the material in the presence of water either:

(i) as an electrolyser when water is normally present in large quantities, or,

(ii) as a fuel cell when product water is produced in the oxygen hydrogen reaction, would be expected to progressively degrade the performance of the material.

#### Conserved Electrolyte (Minimum Liquid Volume) Devices

5 A hydrophilic material as described in the examples above can be used in a Fuel Cell or an Electrolyser provided that the state of hydration of the cell is controlled by only adding that water necessary to the electrolysis reaction or removing only that water produced by the fuel cell reaction. In practice this can be achieved by continuously monitoring the pH of the hydrophilic material  
10 forming the membrane in the working cell.

In electrolyser mode only adding water to prevent the hydrated membrane exceeding the design pH (because the cell can operate either acidic or alkaline it is the modulus pH that is referred to here).

15 In the fuel cell the product water must be removed so that the concentration of TAS is not reduced. This means that the water should preferably be removed by evaporation or 'cold spot' freezing.

**CLAIMS**

1. The use of hydrophilic polymers hydrated in suitable ionic solution as a fuel cell or electrolyser membrane.
2. The use of hydrophilic polymers cast in situ to form an MEA or stack and  
5 hydrated in suitable ionic solution for use in a fuel cell or electrolyser.
3. The design of a minimum liquid volume cell in which the ionically active component is conserved.
4. Any of the above in relation to our one-step manufacture process.
5. Any of the above in which the crosslinked polymer also contains an  
10 ionically active component by crosslinking as defined in GB 2380055A.
6. Any of the above where the ionically active electrolyte is toluene sulphonic acid.
7. Any of the above where the ionically active electrolyte is vinyl sulphonic acid.
- 15 8. Any of the above where the ionically active electrolyte is AMPSA.
9. Any of the above where the ionically active electrolyte is KOH.
10. Any of the above where the ionically active electrolyte is NaOH.



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